## SINTERED BODY AND PRODUCTION METHOD THEREOF

## **BACKGROUND OF THE INVENTION**

[0001] The present invention relates to a powder metallurgy technique for producing a sintered metal body. More particularly, the present invention relates to a sintered metal body having excellent fatigue strength and abrasion resistance to be suitable for use in a sprocket of a silent chain, a high-strength part of an internal combustion engine (e.g. a connecting rod, a piston pin or a rocker arm) or the like and a method for producing such a sintered metal body at low cost.

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[0002] Various techniques have been proposed to increase green density and sintered density for improvement in sinter strength. In one proposed technique, a sintered body is produced by compacting a metal powder material repeatedly and then sintering the compacted powder material repeatedly. In another technique (disclosed in Japanese Laid-Open Patent Publication No. 2001-295915), a sintered body is produced by warm-compacting a metal powder material and sintering the compacted powder material at elevated temperatures.

## **SUMMARY OF THE INVENTION**

[0003] However, the former technique results in relatively high production cost. The latter technique may allow cost reduction, but has a limitation of improvement in sinter strength. For further improvement in sinter strength (notably fatigue strength and abrasion resistance), it is necessary to reduce the size of pores in the sintered body, strengthen particle-to-particle bonding of the sintered body and thereby prevent the occurrence and development of cracking in the sintered body. The proposed techniques are not always effective in pore size reduction and particle-to-particle bond strengthening and are no more than the modifications of the compacting and sintering processes.

[0004] It is therefore an object of the present invention to provide a sintered body having a small pore size, large particle-to-particle contact and strong particle-to-particle bonding so as to prevent crack initiation and

propagation, such that the sintered body attains sufficiently improved fatigue strength and abrasion resistance to be suitable for use in a sprocket of a silent chain, a high-strength part of an internal combustion engine (such as a connecting rod, a piston pin or a rocker arm) etc.

5 [0005] It is also an object of the present invention to provide such a sintered body at relatively low cost.

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[0006] According to a first aspect of the invention, there is provided a sintered body, comprising: sintered metal particles forming a sintered structure and having a maximum particle size of 100  $\mu$ m or smaller; and carbon being dispersed in the sintered structure in an amount of 0.05 to 1.0% by mass based on a total mass of the sintered body.

[0007] According to a second aspect of the invention, there is provided a sintered body having a sintered structure derived from a metal powder and containing carbon dispersed in the sintered structure in an amount of 0.05 to 1.0% by mass based on a total mass of the sintered body, the metal powder having a particle size of 75 µm or smaller.

[0008] According to a third aspect of the invention, there is provided a sintered body produced from a metal powder mixture, the metal powder mixture including a metal powder having a particle size of 75  $\mu$ m or smaller, a graphite powder in an amount 0.1 to 1.0% by mass and a powder lubricant in an amount of 0.05 to 0.80% by mass based on a total mass of the metal powder mixture.

[0009] According to a fourth aspect of the invention, there is provided a production method of a sintered body, comprising: preparing a metal powder mixture, the metal powder mixture including a fine metal powder having a particle size of 75 µm or smaller, a graphite powder in an amount of 0.1 to 1.0% by mass and a powder lubricant in an amount of 0.05 to 0.80% by mass based on a total mass of the metal powder mixture; compacting the metal powder mixture to provide a green compact; and sintering the green compact.

30 [00010] The other objects and features of the invention will also become understood from the following description.

## BRIEF DESCRIPTION OF THE DRAWINGS

[0011] FIG. 1 is a graph showing the abrasion performance of sintered bodies of Examples 1 to 4 according to the present invention.

[0012] FIG. 2 is a graph showing the abrasion performance of sintered bodies of Comparative Examples 1 to 4 according to the earlier technology.

[0013] FIG. 3 is an optical micrograph of the sintered body of Example 3.

[0014] FIG. 4 is an optical micrograph of the sintered body of Comparative Example 3.

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# **DETAILED DESCRIPTION OF THE INVENTION**

10 [0015] The present invention will be described below in detail. In the following description, all percentages (%) are by mass unless otherwise specified.

[0016] As a result of extensive researches, the present inventors have found that the use of a fine powder as a raw material allows substantial reduction of the size of pores in a sintered body in addition to increase in the compactness of a green compact. The present inventors have also found that the use of such a fine powder strengthens particle-to-particle bonding of the sintered body, because the fine powder allows promoted diffusion of powder particles during sintering due to a large particle surface. Although the fine powder is generally low in compactibility, the present inventors have found that the compactibility of the fine powder can be improved by the combination of die lubrication and warm compaction. Further, the present inventors have found that the sintering of the compacted fine powder at a high temperature maximizes the particle diffusion, thereby attaining sinter strength higher than

ever before. The present invention has been made based on the above findings.

[0017] According to the present invention, a sintered body is produced by preparing a metal powder mixture, compacting the metal powder mixture to provide a green compact, and then, sintering the green compact.

The metal powder mixture includes a fine metal powder, a graphite powder and a powder lubricant.

[0019] The fine metal powder usable in the metal powder mixture is not particularly restricted and can be selected among various powder metallurgical materials, such as carbon steel powders and alloyed steel powders, depending on the performance required of the sintered body. In the case of the fine metal powder containing an alloy component, the metal powder may be in the form of a completely alloyed steel powder (prepared by the melting and powdering of a desired steel composition), a partially diffusion-alloyed steel powder (prepared by the diffusion joining of alloying metal grains to iron grains), or a blend of a steel powder and an alloying metal powder optionally together with a partially diffusion-alloyed steel powder. Of various powder metallurgical materials, especially preferred are a blend of an iron-based powder (such as an atomized pure iron powder with an iron content of 90% or more) and an alloying metal powder (such a ferrous alloy powder, a Ni powder, a Cu powder or a Mo powder) and a partially diffusion-alloyed steel powder thereof so as to attain higher compactibility than that attained by a completely alloyed steel powder or the like.

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[0020] Further, the metal powder preferably has a primary particle size of 75  $\mu$ m or smaller. When the primary particles of the metal powder are larger than 75  $\mu$ m in size, the driving force for sintering becomes so weak that that there arise large pores in the sintered body. In the case of using the premix of an iron-based powder and an alloying metal powder (such as a Ni powder a Cu powder or a Mo powder) with a primary particle size greater than 75  $\mu$ m, alloying metal elements do not diffuse properly in the sintering process. As a result, the sintered body cannot be hardened to a sufficient degree even when heat-treated by e.g. gas carburizing, bright annealing or induction hardening for further improvement in strength, so that the sintered body has a relatively soft and rough ferrite or pearlite structure that can result in fatigue failure. The fatigue strength of the sintered body thus becomes low.

[0021] Desirably, the metal powder has been granulated using a binder (such as alumina sol or water glass), or by diffusion joining, so as to agglomerate the primary particles into secondary particles having a particle

size of 180  $\mu m$  or smaller. The granulation of the metal powder increases the apparent particle size and fluidity of the metal powder. As the mechanical properties of the sintered body depends on the primary particle size of the raw material, the fatigue strength of the sintered body does not become lowered as compared with a case where the metal powder has not been granulated. When the secondary particles of the metal powder are larger than 180  $\mu m$  in size, the packability of the metal powder into a thin area becomes low.

The metal powder can be classified by screening for control of the primary and secondary particle sizes. Namely, the primary particle size of the metal powder can be controlled to 75  $\mu$ m or smaller by, before the granulation process, passing the metal powder through a screen mesh having a mesh size of 75  $\mu$ m (200 mesh) as defined by JIS Z8801. Further, the secondary particle size of the metal powder can be controlled to 180  $\mu$ m or smaller by passing the granulated metal powder through a screen mesh having a mesh size of 180  $\mu$ m (80 mesh) as defined by JIS Z8801.

[0023] The graphite powder is contained in the metal powder mixture in an amount of 0.05 to 1.0% based on the total mass of the metal powder mixture, such that graphite becomes dispersed throughout the sintered body for solid-solution strengthening. When the amount of graphite powder exceeds 1.0%, the green compact becomes low in density. When the amount of graphite powder is less than 0.1%, the graphite powder fails to provide a sufficient solid-solution strengthening effect.

The powder lubricant is contained in the metal powder mixture in an amount of 0.05 to 0.80% based on the total mass of the metal powder mixture, so as to increase the mobility of the solid particles of the metal powder mixture and thereby improve the compactibility of the metal powder mixture. When the amount of powder lubricant is less than 0.05%, the powder lubricant fails to provide sufficient lubricity so that the green compact becomes low in density. In addition, the compact is susceptible to green cracking. When the amount of powder lubricant exceeds 0.80%, the powder lubricant keeps the powder mixture from plastic deformation rather than

provides lubricity. The density of the green compact thus becomes so low that the sintered body decreases in density.

[0025] The powder lubricant usable in the metal powder mixture is not particularly restricted and can be selected from various lubricating materials to be released in the sintering process. Specific examples of the powder lubricant include metallic soaps such as zinc stearate, lithium stearate and calcium stearate, and waxes such as ethylene-bis-stearoamide. These lubricant compounds can be used alone or in any combination thereof.

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[0026] The metal powder mixture is desirably compacted while being heated to e.g. 100°C or higher, so as to densify the green compact and thereby reduce the size of pores between the particles and increase contact between the particles. When the temperature of the metal powder mixture in the compacting process is too high, there is a possibility that the powder lubricant melts to deteriorate the fluidity of the powder mixture. The highest temperature of the metal powder mixture in the compacting process is thus preferably limited to 150°C.

[0027] If the heated metal powder mixture is compressed into an unheated die, the metal powder mixture becomes cooled. In addition, there occurs heat transfer from the metal powder mixture to the die by the time the compacting process is completed. In view of such circumstances, the die is preferably preheated to a temperature higher than the temperature of the metal powder mixture, e.g. 120°C or higher.

[0028] Further, the metal powder mixture is desirably compacted with a die lubricant applied to an inner surface of the die, so as to minimize the amount of powder lubricant contained in the metal powder mixture and thereby increase the density and strength of the sintered body. The die lubricant usable is basically the same as the powder lubricant, such as zinc stearate, lithium stearate and calcium stearate or ethylene-bis-stearamide. In the case of applying the die lubricant by an electrostatic method, the die lubricant needs to be selected in view of its electrostatic property.

[0029] The process of applying the die lubricant to the die is not

particularly restricted. For example, the die lubricant can be electrostatically adhered to the die by injecting a charged solid powder of die lubricant into the The amount of die lubricant applied to the die is desirably 5 to  $100 \text{ g/m}^2$ . When the amount of die lubricant is less than 5 g/m<sup>2</sup>, the above-mentioned effects cannot be obtained due to inadequate die lubrication. Moreover, the force to remove the compact from the die becomes large. When the amount of die lubricant exceeds 100 g/cm<sup>2</sup>, the die lubricant remains on a surface of the green compact, thereby resulting in deteriorated appearance.

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[0030] The green compact is desirably sintered at a temperature of 1180°C or higher in an endothermic gas atmosphere (RX atmosphere), a hydrogen-containing nitrogen atmosphere or an ammonolysis atmosphere, or in a vacuum. In combination with close packing of the metal powder mixture in the compacting process, the sintering of the green compact at such a high temperature effectively promote the diffusion of the particles in the sintering process so as to form stronger bonding between the particles and to densify the sintered body. However, the cost of sintering increases with increase in the sintering temperature. It is thus necessary to select the sintering temperature appropriately for strength/cost tradeoffs.

The sintered body may be given any heat treatment as needed so as to increase in surface hardness and thereby obtain a further improvement in The heat treatment is not particularly restricted and can be a known treatment, such as gas carburizing, bright annealing or induction hardening. [0032] The thus-produced sintered body has a sintered structure derived from the fine metal powder, i.e., a coherent bonded structure formed of sintered

metal particles. (The "sintered metal particles" are herein defined as including agglomerates of pluralities of powder particles formed in the sintering process.)

[0033] The sintered metal particles have a maximum particle size of 100 μm or smaller. (Herein, the "maximum particle size" refers to the largest diameter of the particles.) When the maximum size of the sintered metal particles is larger than 100  $\mu$ m, the pore size of the sintered body is too large so that the initial defect of the sintered body that can result in exfoliation abrasion increases in size. The sintered body thus becomes low in strength and abrasion resistance.

[0034] Further, the sintered body contains 0.05 to 1.0% of carbon derived from the graphite powder and carbon of the metal powder and dispersed in the sintered structure, based on the total mass of the sintered body. It is noted that a maximum of 0.05% of carbon is burned down in the sintering process. When the amount of carbon in the sintered body is less than 0.05%, the sintered body cannot attain desired strength and abrasion resistance. When the amount of carbon in the sintered body exceeds 1.0%, the sintered body is so hard as to be susceptible to sintering crack and embrittlement.

[0035] It is therefore possible in the present invention to reduce the pore size of the sintered body and to maximize the particle-to-particle contact and particle-to-particle bonding of the sintered body, thereby preventing the pores from being linked together to cause cracking in the sintered body. The produced sintered body advantageously shows excellent fatigue strength and abrasion resistance to be fit for a wide range of uses, such as a sprocket of an silent chain and a high-strength part of an internal combustion engine (e.g. a connecting rod, a piston pin or a rocker arm).

[0036] Especially, teeth of the sprocket are susceptible to abrasion as the sprocket teeth receive, from the silent chain, a high surface pressure and a large impactive load accompanied by slippage. The abrasion of the sprocket teeth is classified as "exfoliation abrasion" in which cracking occurs and develops due to the link-up of pores in the sprocket to cause exfoliation in a surface of the sprocket teeth. It becomes however possible to effectively protect the sprocket from such exfoliation abrasion by applying the above sintered body to the sprocket, because the sintered body is small in pore size, large in particle-to-particle contact and high in particle-to-particle bond strength. For cost reduction, the above sintered body may be applied only to the teeth of the sprocket while applying a low-priced material to other portions of the sprocket. For example, the sprocket may be produced by classifying a powder metal into

a fine powder having a primary particle size of 75  $\mu$ m or smaller and a residual powder, and then, using the fine powder for the teeth of the outer peripheral portion of the sprocket while using the residual powder for the inner peripheral portion of the sprocket, thereby reducing the cost of raw materials.

[0037] By applying the above sintered body to the high-strength part of the internal combustion engine (e.g. connecting rod, piston pin or rocker arm), it becomes possible to attain not only cost reduction but also weight reduction of the engine part. The input of inertia into a crankshaft journal decreases with the weight of the engine part. The fuel economy of the engine can be thus improved successfully.

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[0038] The present invention will be described in more detail by reference to the following examples. However, it should be noted that the following examples are only illustrative and not intended to limit the invention thereto.

# 1. Rotating Bending Fatigue Test

[0039] 15 Each test sample was produced by the following procedure. [0040] A partially diffusion-alloyed steel powder (as a fine metal powder) comprised of 4% of Ni, 0.5% of Mo, 1.5% of Cu and the balance being Fe was mixed with a graphite powder and ethylene-bis-stearamide (as a powder lubricant) by the use of a V-blender, thereby providing a metal powder mixture as a raw material. Herein, the partially diffusion-alloyed steel powder had 20 been formed by classifying an atomized pure iron powder by screening, blending the classified iron powder with a powder of Ni, Mo and Cu (having a particle size of 1 to 10 µm), heat-treating the blend at 850°C in such a manner as to adhere alloying metal elements of Ni, Mo and Cu to iron grains by diffusion joining, and then, classifying the resultant granulated powder by 25 screening. The maximum primary and secondary particle sizes of the metal powder and the amounts of graphite powder and powder lubricant added in the metal powder mixture were controlled as indicated in TABLE.

[0041] Subsequently, a die was preheated to a given temperature. Then, the metal powder mixture was heated to a given temperature and compacted in the preheated die by the application of a pressure of 686 MPa (7 t/cm<sup>2</sup>), thereby

forming a green compact with a length of 80 mm, a width of 15 mm and a height of 15 mm. Prior to the compacting process, zinc stearate (as a die lubricant) was electrostatically adhered to an inner surface of the die in Examples 2 and 3 and Comparative Examples 2 to 4. The amount of die lubricant applied to the die was 10 g/m<sup>2</sup>. The green compact was sintered at a given temperature for 1 hour in an N<sub>2</sub> atmosphere containing 10 vol% of H<sub>2</sub>, thereby providing an iron-based sintered compact. In each example, the heat temperatures of the metal powder mixture and the die in the compacting process and the sintering temperature were controlled as indicated in TABLE.

10 [0042] A test sample having a parallel portion diameter of 8 mm and a straight portion length of 15.4 mm was cut from the sintered compact and then heat-treated under the following conditions.

(Heat-Treatment Conditions)

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Example 1 and Comparative Example 1: subjected to bright annealing, i.e.,

heated at 900°C for 1 hour, oil-quenched at 60°C, and then, tempered at 180°C for 1 hour.

Examples 2 to 4 and Comparative Examples 3 and 4: subjected to gas carburizing, i.e., heated at 900°C for 2 hours in a carburizing atmosphere with a carbon potential of 0.9%.

20 The test sample of Comparative Example 2 was given no heat treatment.

[0043] The thus-produced test samples of Examples 1 to 4 and Comparative Examples 1 to 4 were subjected to rotating bending fatigue test according to JIS Z 2274. In the test, the fatigue limit was defined as 10<sup>7</sup> cycles and evaluated as a rotating bending fatigue strength. The test results are shown in TABLE.

[0044] Further, the densities of the green compacts and the heat-treated sintered compacts of Examples 1 to 4 and Comparative Examples 1 to 4 were measured by so-called Archimedes method, in which the volume of a measuring object is calculated by immersing the measuring object in ethanol.

30 The test results are shown in TABLE.

[0045] For observation by an optical microscope, the heat-treated sintered

compacts of Example 3 and Comparative Examples 3 and 4 were cut in half, and then, the resultant sample pieces were embedded in resinous sample stages and polished. The thus-obtained samples were microscopically examined to observe sintered structures of Example 3 and Comparative Examples 3 and 4 and determine a maximum size of the sintered metal particles of each sintered structure in a observation field of 63 mm × 92 mm. The results are shown in TABLE and FIGS. 3 and 4.

## 2. Abrasion Resistance Test

[0046] In each of Examples 1 to 4 and Comparative Examples 1 to 4, a sprocket having 42 involute gear teeth (tooth width: 8.4 mm, over-pin diameter: 84.88 mm, gauge-pin diameter: 3.492 mm) was produced in the same manner as in the production of the above test sample. The produced sintered sprocket was attached to a camshaft of an in-line four-cylinder, 1.8-liter gasoline engine with another sprocket attached to a crankshaft of the engine.

The sintered sprocket and the other sprocket were linked by a 5- by 4-row silent chain (pitch: 6.35 mm, effective width: 10.9 mm), and then, the crankshaft was driven by an electric motor under the following conditions to examine an abrasion loss of the teeth of the sprocket of each example. The test results are indicated in FIGS. 1 and 2. In FIGS. 1 and 2, the abrasion loss of the sprockets of Examples 2 to 4 and Comparative Examples 1 to 4 are indicated with respect to the abrasion loss of the sprocket of Example 1 as being represented by 1 (one).

(Test Conditions)

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Tension of Silent Chain: 1500 N (one sided tensioning)

25 Engine Speed (Crankshaft Rotational Speed): 6000 rpm

Test Time: 100 hours

Lubricant: 5W30SG

Lubricant Temperature: 110°C

TABLE

Green density (g/cm <sup>2</sup> )			7.23	7.38	7.38	6.99	7.27	7.34	7.38	6.90
Die lubrication			none	done	done	none	none	done	done	done
cting ature )		Die	120	130	130	25	120	110	130	100
Compa	Compacting temperature (°C)		100	120	120	25	110	100	120	06
	Lubricant amount (%)		0.6	0.2	0.3	0.5	9.0	0.1	0.2	1.0
Metal powder mixture	Graphite amount (%)		6.0	6.0	6.0	6'0	6.0	6'0	0.3	2.0
Metal pow	Maximum particle size (µm)	Secondary particle	45	106	180	100	180	180	180	100
		Primary particle	45	63	75	70	180	180	180	100
			Example 1	Example 2	Example 3	Example 4	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4

	Fatigue strength (MPa)		450	260	550	440	380	300	470	400
TABLE (continued)		Maximum particle size (µm)	(-)	(–)	80	(_)	(-)	(_)	150	130
	Sintered body	Carbon content (%)	0.87	0.28	0.27	0.85	0.88	0.85	0.28	99:0
		Sintered density (g/cm²)	7.38	7.53	7.50	7.01	7.34	7.41	7.45	7.01
		Heat	bright annealing	carburizing	carburizing	carburizing	bright annealing	none	carburizing	carburizing
		Sintering temperature (°C)	1250	1250	1250	1130	1250	1250	1250	1250
			Example 1	Example 2	Example 3	Example 4	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4

[0047] In each of Examples 1 to 4, the metal powder mixture was prepared by mixing the metal powder having a primary particle size of 75 µm of smaller and a secondary particle size of 180 µm or smaller with the above-specific amounts of graphite powder and powder lubricant, compacted and then The sintered metal particles of the resultant sintered body of Example 3 had a maximum particle size of 100 µm or smaller, although the primary particle size of the metal powder of Example 3 was larger than those of Examples 1, 2 and 4. It is estimated that the sintered metal particles of the sintered bodies of Examples 1, 2 and 4 also had a maximum particle size of 100 µm or smaller in consideration of the interrelationship between the primary particle size and the maximum sintered metal particle size. Accordingly, the sintered bodies of Examples 1 to 4 had high fatigue strength and abrasion resistance as indicated in TABLE and FIGS. 1 and 2. The temperature of the metal powder mixture in the compacting process was lower in Example 4 than in Examples 1 to 3, and die lubrication was not performed in Example 4. This resulted in lower green density and sintered density. The sintered body of Example 4 however had high fatigue strength and abrasion resistance, benefiting from the effect of using the above-mentioned fine metal powder as a raw material.

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[0048] On the other hand, the metal powders of Comparative Examples 1 and 2 had a primary particle size larger than 75  $\mu m$ . On the analogy of Comparative Example 3, it is estimated that the sintered metal particles of the sintered bodies of Comparative Examples 1 and 2 had a maximum size exceeding 100  $\mu m$ . The sintered bodies of Comparative Examples 1 and 2 were therefore low in fatigue strength and abrasion resistance. The metal powder of Comparative Example 4 also had a primary particle size larger than 75  $\mu m$ . Further, the amount of powder lubricant added in the metal powder mixture was too much in Comparative Example 4. This resulted in low sintered density and a maximum sintered metal particle size exceeding 100  $\mu m$ . Thus, the sintered body of Comparative Example 4 was also low in fatigue

strength and abrasion resistance. The sintered body of Comparative Example 3 had a maximum sintered metal particle size larger than  $100~\mu m$ . The fatigue strength of the sintered body of Comparative Example 3 was thus similar to those of the low-density sintered bodies of Comparative Examples 1, 2 and 4, although the sintered body of Comparative Example 3 was relatively high in density. Further, the abrasion resistance of the sintered body of Comparative Example 3 was low.

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[0049] As described above, the sintered body can be produced by preparing the metal powder mixture from the fine metal powder, the graphite powder and the powder lubricant, warm-compacting the metal powder mixture optionally with die lubrication, and then, sintering the compacted powder mixture at a relatively high temperature in the present invention. If necessary, the metal powder may be granulated to a desired particle size. This makes it possible to provide the sintered body with a very small pore size, maximized particle-to-particle contact, strong particle-to particle bonding and high sintered density, thereby effectively preventing the occurrence and development of cracking in the sintered body effectively. In addition, there is no need to repeat the compaction and sintering processes for increased green density and sintered density. The sintered body is therefore produced at relatively low cost while attaining significantly improved fatigue strength and abrasion resistance to be suitable for use in a sprocket of a silent chain or a high-strength part of an internal combustion engine.

[0050] The entire contents of Japanese Patent Application No. 2003-009483 (filed on January 17, 2003) are herein incorporated by reference.

[0051] Although the present invention has been described with reference to specific embodiments of the invention, the invention is not limited to the above-described embodiments. Various modification and variation of the embodiments described above will occur to those skilled in the art in light of the above teaching. The scope of the invention is defined with reference to the following claims.